ROPUBLIC OF SOUTH AFRICA REVENUE PATENTS ACT, 1978 FORM P.1 APPRICATION FOR A PATENT AND (to be lodged in duplicate) ACKNOWLEDGEMENT OF RECEIPT <del>-</del>7. 3.91 130,00 (Section 30(1) Regulation 23) REPUBLIEK VAN SUID AFRIKA THE GRANT OF A PATENT IS HERBBY REQUESTED BY THE UNDERMENTIONED APPLICANT ON THE EASIS OF THE PRESENT APPLICATION FILED IN DUPLICATE. PATENT APPLICATION NO. A & A REF: 122017 AS/CST 688 FULL NAME(S) OF APPLICANT(S) CSIR Address(es) of applicant(s Corporate Building, Scientia, PRETORIA, Transvaal Province, Republic of South Africa 54 TITLE OF INVENTION BINDER Only the items marked with an "X" in the blocks below are applicable. THE APPLICANT CLAIMS PRIORITY AS SET OUT ON THE ACCOMPANYING FORM P.2 THE APPLICATION IS FOR A PATENT OF ADDITION TO PATENT APPLICATION NO. 21 01 THIS APPLICATION IS A FRESH APPLICATION IN TERMS OF SECTION 37 AND BASED CN APPLICATION NO. THIS APPLICATION IS ACCOMPANIED BY: 1. A SIRE SON OF PRINCIPAL two copies of a complete specification of .... 15... pages
2. Drawings of ...... aheets, 3. Publication particulars and abstract (Form P.S in duplicate) (for complets only). A copy of Figure . . . . of the drawings (if any) for the abstract (for complete only). An assignment of invention. Certified priority document(s) (State quantity) : Translation of the priority document(s). An assignment of priority rights. A copy of the Form P.2 and the specification of S.A. Patent Application No. 10. A Form P.2 in duplicate. 90/2226 11. A declaration and power of attorney on Form P.3. 12. Request for ante-dating on Form P.4. 13. Request for classification on Form P.9. 区 14. 15. 14. Request for delay of acceptance on Form P.4. ADDRESS FOR SERVICE: Adams & Adams, Pretoria. DATED THIS 7th DAY OF March 19 91 TEMTS, DEBISNS, REGISTIVE PECETVENT THEM ADAMS & ADAMS APPLICANTS PATENT ATTORNEYS TE STAMP The duplicate will be returned to the applicant's address for service as proof of lodging but is not valid unless endorsed with official stamp. REGISTRAN OF PATENTS

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ADAMS & ADAMS PATENT ATTORNEYS SHORBURG PRETORIA

BINDER

FORM P7

REPUBLIC OF SOUTH AFRICA Patents Act, 1978

# COMPLETE SPECIFICATION

(Section 30 (1) - Regulation 28)

OFFICIAL APPLICATION	NO.		LODGING DATE
21 01 91	1688	<del>-</del>	22 7 March 199
INTERNATIONAL CLASSIF	IGATION		
51 GO4B B28B B2	86		
FULL NAMES(8) OF APPL	ICANT(S)		
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THIS INVENTION relates to a binder. More particularly, it relates to a binder suitable for use as a substitute for ordinary portland cement.

According to one aspect of the invention there is provided a binder which comprises a Class F fly ash and an alkali metal compound selected from the group consisting of alkali metal hydroxides, alkali metal silicates and mixtures thereof, the binder being in the form of a particulate mixture comprising, for every 100 parts by mass of fly ash, 1 - 40 parts by mass of said alkali metal compound.

By Class F fly ash is meant fly ash classified by the American Society for Testing and Materials (ASTM) under Standard Specification Designation C618-87 as Class F fly ash. Typical chemical compositions of Class F fly ash available in South Africa are as set forth in the following table, Table 1:

TABLE 1

Constituent	% by Mass
sio <sub>2</sub>	40,04 - 50,6
Al <sub>2</sub> O <sub>3</sub>	26,30 - 38,93
Fe <sub>2</sub> O <sub>3</sub>	2,63 - 5,55
CaO	4,42 - 12,80
MgO	
so <sub>3</sub>	1,15 - 3,10
Na <sub>2</sub> O	0,16 - 1,13
K <sub>2</sub> O	0,30 - 1,03
- 1	0,20 - 0,99
Loss upon Ignition	0,80 - 3,86

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Optionally, the binder comprises a proportion, eg. 1-50 parts by mass of amorphous silica, for every 100 parts by mass of said fly ash. The amorphous silica may be fumed silica condensate or so-called silica fume.

Preferred proportions of the constituents depend to a degree on the nature of the constituents and the use for which the binder is intended. Thus, when the binder is intended for use in making cast products and the alkali metal compound is a sodium compound, the binder may contain 5 - 40 parts by mass of the sodium compound and up to 50 parts by mass of amorphous silica, for every 100 parts by mass of fly ash; when the binder is intended for use in making cast products and the alkali metal compound is a potassium compound, the binder may contain 7 - 40 parts by mass of the potassium compound and up to 50 parts by mass of amorphous silica, for every 100 parts by mass of fly ash; and when the binder is intended for use in making pressed products, it may contain 1 - 20 parts by mass of the alkali metal compound and up to 50 parts by mass of amorphous silica, for every 100 parts by mass of fly ash. For reasons of cost and availability, as indicated above, sodium and potassium alkali metal compounds are preferred.

More particularly, if the alkali metal compound is a hydroxide, then the preferred proportions depend on whether amorphous silica such as silica fume is added or not. Table 2 below indicates the preferred proportions of the different constituents on a parts by mass basis.

TABLE 2 (NO SILICA FUME)

	7	<u></u>
Alkali Metal in Question	Fly Ash Proportion	Preferred Alkali Metal Hydroxide Proportion
Potassium	100	Froportion
Sodia	100	12 - 25
Sodium	100	6 - 25
		6 ~ 25

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Better strengths are obtained when silica fume is added. to the mix with the best strengths obtained when the silica fume is first dissolved in the hydroxide solution made by mixing the required alkali metal hydroxide with the required. amount of water before mixing in the fly ash. The preferred ranges of the different components are represented in Table 3

TABLE 3 (AMORPHOUS SILICA ADDED)

Alkali Metal .P in Question P	ly Ash roportion	Alkali Metal	Amount
	•	Hydroxide Proportion	Amorphous Silica (Fumed Silica Condensate)
Potassium	100	8 - 36	Proportion 6 - 40
Sodium	100	5 - 30	10 - 50

It is also possible to use commercially available alkali metal silicate solutions or soluble powders, or mixtures of alkali metal silicates and alkali metal hydroxides, rather than mixtures of alkali metal hydroxides and amorphous silica. The preferred proportions for potassium and sodium silicates are presented in Table 4 below. It should be noted that the silicate proportions are presented on an anhydrous basis.

TABLE 4

Alkali Metal in		<del>-</del>
Question	Fly Ash Proportion	
Potassium		Silicate Proportion
<u>li</u>	100	10 - 40
Sodium	100	~ · · · · · · · · · · · · · · · · · · ·
		19 - 40

The amount of water used in combination with the other constituents affects the strength of the binder. Generally,... the higher the water content the weaker the strength of the

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binder. However in the case of cast products a minimum water content is required in order for the mix to be workable and therefore to be compacted properly.

The fly ash and alkali metal compound may together form at least 66% by mass of the binder, preferably at least 80%.

Typically the binder will be used in a fashion similar to that of ordinary portland cement. Thus, it will be thoroughly mixed with water and optionally with a particulate filler or diluent such as granular particles (eg. sand, additional fly ash or granulated slag), fibres (eg. glass, steel, organic polymers or asbestos), and/or aggregate (gravel or stone), after which it is cast, pressed, moulded or extruded and then caused or allowed to set.

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With regard to these fillers or diluents, the proportions 15 ° thereof used will depend on what degree of shrinkage or expansion can be tolerated and/or the strength required in the eventual set product. For mortars and concretes the binder:filler/diluent (sand or stone) mass ratio may be 1:1 -1:9 for continuously graded fillers or diluents; and as a first approximation proportions, particle sizes and/or particle size distributions will be employed which are similar to those employed in portland cement/concrete practice.

The invention extends to a method of making an artifact which method comprises admixing a binder as described above with water, shaping the mixture into a green artifact of a desired shape, and causing or allowing the green artifact to set, 8 - 200 parts by mass of water being admixed with the binder for every 100 parts by mass of the fly ash in the

30 The shaping may be by casting, 20 - 200 parts by mass of water being admixed with the binder for every 100 parts by mass of fly ash in the binder, the alkali metal compound of the binder being selected from sodium compounds, potassium

compounds and mixtures thereof. Instead, the shaping may be by pressing, 8 - 20 parts by mass of water being admixed with the binder for every 100 parts by mass of the fly ash.

The green artifact may be caused to set by maintaining it at an elevated temperature of 40 - 100°C, in eg a conventional oven or a microwave oven for a period of a few minutes up to several, eg 4, days, with the artifact enclosed in a water-impermeable enclosure such as a plastics or similar water-impermeable membrane, or being located under water to prevent it from drying out prematurely. This curing at elevated temperature leads to enhanced early strength after 1 - 4 days. The green artifact can instead be allowed to set at an ambient temperature of not more than 40°C, while being maintained in a moist condition, but strength development thereof is somewhat slower.

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As indicated above, the method may comprise admixing a particulate filler or diluent with the binder and water. The nature of the diluent or filler used and the proportions thereof will depend on the artifact to be made, examples being 8 - 20 parts by mass of asbestos fibre for every 100 parts binder for fibre-reinforced sheets; and 100 - 900 parts by mass of clean sand for every 100 parts binder for general purpose mortar applications. In general, concrete practice when employing portland cement can be used as a guide in this regard.

The invention also extends to a method of making an artifact which comprises admixing fly ash, an alkali metal compound selected from the group consisting of alkali metal hydroxides, alkali metal silicates and mixtures thereof, and water, shaping the mixture into a green artifact and causing or allowing the artifact to set, 1 - 40 parts by mass of said alkali metal compound being admixed with every 100 parts by mass of fly ash.

If the alkali metal compound and fly ash are not premixed into a binder as described above, the alkali metal compound may first be dissolved in the water to form a solution, after which the fly ash is admixed into the solution. If amorphous silica is used, it may be admixed with the solution before the fly ash, any other constituents being admixed into the solution after the fly ash.

The invention extends further to a binder which comprises fly ash and an alkali metal compound selected from the group consisting of alkali metal hydroxides, alkali metal silicates and mixtures thereof, the fly ash and alkali metal compound together making up at least 66% by mass of the binder, preferably at least 80%.

The invention extends still further to an artifact

whenever made in accordance with the method described above.

The invention will now be described, by way of illustration, with reference to the following non-limiting Examples, in which the fly ash used was a Class F fly ash received from the Matla Power Station having the following chemical analysis:

Constituent	% by Mass
Sio <sub>2</sub>	46,60
A1 <sub>2</sub> 0 <sub>3</sub>	33,86
Fe <sub>2</sub> O <sub>3</sub>	4,22
Cao	9,31
MgO	2,20
so <sub>3</sub>	0,76
Na <sub>2</sub> o	0,52
K <sub>2</sub> O	0,20
Loss upon Ignition	1,54

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The fly ash had a particle size such that 6% by mass thereof was retained on a test sieve of 0,045 mm nominal size, a specific surface of 3400 cm $^2$ /g and a density of 2,31 (ie g/cm $^3$ ).

The sand used was clean quartz sand from Philippi, Cape Province, having a particle size such that it all passed through a test sieve of 0,850 mm nominal size and not more than 10% by mass thereof passed through a test sieve of 0,600 mm nominal size.

The fumed silica condensate used was obtained from a ferrosilicon plant in Pietersburg and had the following chemical analysis:

Mass
94,06
0,72
0,08
0,13
0,47
0,42
0,23
0,42

The funed silica had a relative density of 2,27 and a specific surface of 37 000  ${\rm cm}^2/{\rm g}$ .

The sodium hydroxide and potassium hydroxide used were of laboratory grade and were in the form of pellets.

SABS (South African Bureau of Standards) Method No. 866 was followed in the making of mortar specimens. Mixing was

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effected by a mechanical mixer as described in SABS Method No. 866. The green mix was placed in steel prism moulds and compacted as described in SABS Method No. 866. After compaction the moulds were inserted into plastics bags having a volume of 10 litres which were then sealed to prevent moisture from escaping. Unless stated otherwise the green mix was cured at 40°C for a predetermined period, after which a cured or set artifact was obtained, whose strength was tested in conventional fashion using methods used to test cement or concrete artifacts. An Instron testing machine was used to determine the flexural strength of all the artifacts. For testing compressive strength said Instron machine was used except for artifacts with a compressive strength of over 40 MPa, in which case a Baldwin testing machine was used.

In all examples the proportions are given on a mass basis.

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#### Example 1

A binder composition comprising 20,2 parts potassium hydroxide for every 100 parts fly ash was used together with 30 parts of water and 300 parts of sand. After two days curing the resulting artifact, which amounted to a mortar, had a compressive strength of 1,69 MPa, which increased after 9 days to 14,75 MPa, and after 26 days to 23,0 MPa. The equivalent flexural strengths were respectively 0,7 MPa, 4,1 MPa and 6,3 MPa.

#### Example 2

In this case, a binder composition comprising 28,21 parts potassium hydroxide and 15,09 parts amorphous silica (fumed silica condensate) for every 100 parts fly ash was used together with 38,4 parts water and 345 parts sand. In this case, after 2, 9 and 26 days respectively, a mortar artifact was obtained whose compressive strengths were 2,27 MPa, 22,66 MPa

and 25,0 MPa. The equivalent flexural strengths after the same periods were respectively 0,8 MPa, 4,6 MPa and 6,3 MPa.

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#### Example 3

In this case a binder comprising 9,05 parts sodium hydroxide for every 100 parts fly ash was used together with 38,89 parts water and 300 parts sand. In this case after 2, 7 and 26 days respectively, a mortar artifact was obtained whose compressive strengths were 2,47 MPa, 24,34 MPa and 33,0 MPa, the equivalent flexural strengths after the same period respectively being 0,7 MPa, 4,5 MPa and 6,5 MPa.

## Example 4

In this case the binder was not pre-mixed, but its various constituents were mixed separately to make up the green mix. For every 100 parts fly ash used, there were used 14,25 parts sodium hydroxide, 21,50 parts fumed silica condensate, 44 parts water and 360 parts sand. The sodium hydroxide was first dissolved in the water and then the fumed silica condensate was added to the solution and dissolved therein with mixing. The resulting silicate solution became very hot and was left to cool until it reached ambient temperature. The solution was then thoroughly blended and mixed with the remaining constituents to form the green mixture. In this case, after one day a mortar artifact was obtained which had a compressive strength of 11,6 MPa, which increased after 7 days to 50,9 MPa, and after 14 days to 64,2 MPa. After the same periods the respective equivalent flexural strengths were 2,3 MPa, 6,8 MPa and 8,6 MPa.

#### EXAMPLE 5

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In this case, a binder composition comprising 8,19 parts sodium hydroxide and 11,62 parts amorphous silica (fumed silica condensate) for every 100 parts fly ash was used together with 30,21 parts water and 335 parts sand. The sodium hydroxide was first dissolved in water and then the fumed silica condensate was added to the solution and dissolved therein with mixing. The resulting silicate solution became very hot and was left to cool until it reached ambient temperature. The solution was then thoroughly blended with the fly ash and then the sand was added. In this case, after two days the mortar artifact obtained a compressive strength of 31,44 MPa, which increased after 7 days to 51.56 MPa and after 14 days to 51,63 MPa. After the same periods the respective equivalent flexural strengths were 5,05, 7,14 and 6,25 MPa respectively.

#### EXAMPLE 6

In this case, a binder composition comprising 12,22 parts sodium hydroxide and 22,93 parts amorphous silica (fumed silica condensate) for every 100 parts fly ash was used together with 43,47 parts water and 178,16 parts sand. The sodium hydroxide was first dissolved in water and then the fumed silica condensate was added to the solution and dissolved therein with mixing. The resulting silicate solution became very hot and was left to cool until it reached ambient temperature. The solution was then thoroughly blended with the fly ash and then the sand was added. In this case the specimens were cured at room temperature. The flexural and compressive strengths at different times are given

Time (Days)	Flexural Strength (MPa)	Comressive
1 2	0,9	Strength (MPa)
14 21	3,8 4,9	4,3 16,8 28,0
28	6,7	37,7 35,8

## EXAMPLE 7

The soluble sodium silicate used in this Example was obtained from Silicate and Chemical Industries, as so-called Silchem SP20 grade, with a reported SiC2:Na2O ratio of 2:1 and a mean total solids content of 80% by mass.

In this case, a binder composition comprising 30,19 parts sodium silicate for every 100 parts fly ash was used together with 45,33 parts water and 360,00 parts sand. The sodium silicate was first dissolved in water and the solution was then thoroughly blended with the fly ash and then the sand was added. In this case, after two days the mortar artifact obtained a compressive strength of 9,88 MPa, which increased after 7 days to 23,34 MPa, and after 26 days to 34,28 MPa. After the same periods the respective equivalent flexural strengths were 2,83, 4,45 and 5,67 MPa respectively.

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- 1. A binder which comprises a Class F fly ash and an alkali metal compound selected from the group consisting of alkali metal hydroxides, alkali metal silicates and mixtures thereof, the binder being in the form of a particulate mixture comprising, for every 100 parts by mass of fly ash, 1 40 parts by mass of said alkali metal compound.
- 2. A binder as claimed in claim 1, which comprises 1-50 parts by mass of amorphous silica for every 100 parts by mass of said fly ash.
- 3. A binder as claimed in claim 1 or claim 2, in which the fly ash and elkali metal compound together form at least 66% by mass of the binder.
- 4. A method of making an artifact, which method comprises admixing a binder as claimed in claim 1 with water, shaping the mixture into a green artifact of a desired shape, and causing or allowing the green artifact to set, 8 200 parts by mass of water being admixed with the binder for every 100 parts by mass of fly ash in the binder.
- 5. A method as claimed in claim 4, in which the shaping is by casting, 20-200 parts by mass of water being admixed with the binder for every 100 parts by mass of the fly ash in the binder, the alkali metal compound of the binder being selected from sodium compounds, potassium compounds and mixtures thereof.

- 6. A method as claimed in claim 4, in which the shaping is by pressing, 8-20 parts by mass of water being admixed with the binder for every 100 parts by mass of the fly ash.
- 7. A method as claimed in any one of claims 4-6 inclusive, in which the green artifact is caused to set by maintaining it an elevated temperature of 40-100°C with the artifact enclosed in a water-impermeable enclosure or located under water.
- 8. A method as claimed in any one of claims 4-7 inclusive
  10 which comprises admixing a particulate filler or diluent with
  the binder and water.
- 9. A method of making an artifact which comprises admixing fly ash, an alkali metal compound selected from the group consisting of alkali metal hydroxides, alkali metal silicates and mixtures thereof, and water, shaping the mixture into a green artifact and causing or allowing the artifact to set, 1-with every 100 parts by mass of fly ash.
- 10. A method as claimed in claim 9, in which the alkali metal compound is first dissolved in the water to form a solution, after which the fly ash is admixed into the solution.
  - 11. A method as claimed in claim 10, in which amorphous silica is admixed into the solution before the fly ash, any other constituents being admixed into the solution after the fly ash.

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12. A binder which comprises fly ash and an alkali metal compound selected from the group consisting of alkali metal hydroxides, alkali metal silicates and mixtures thereof, the fly ash and alkali metal compound together making up at least 66% by mass of the binder.

- 13. A binder, substantially as described herein.
- 14. A method of making an artifact, substantially as

DATED THIS 7TH DAY OF MARCH, 1991

ADAMS & ADAMS APPLICANTS PATENT ATTORNEYS

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